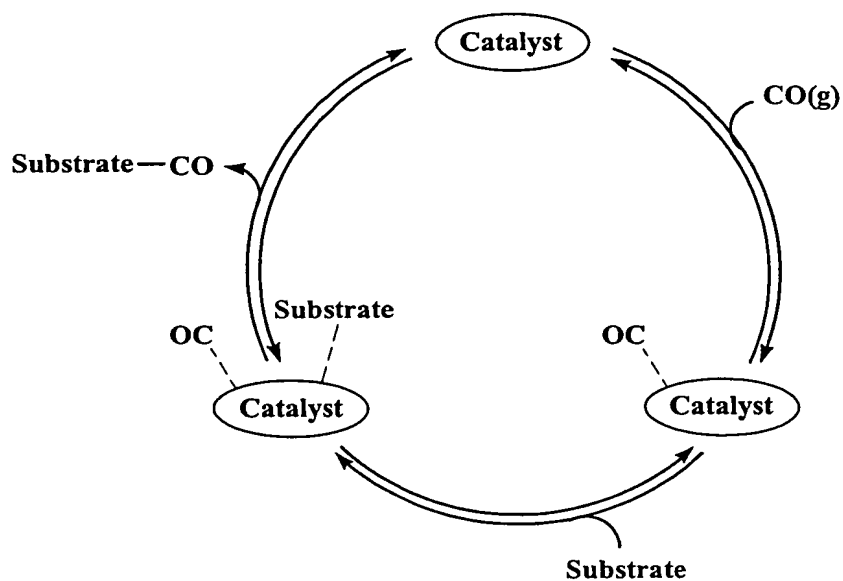




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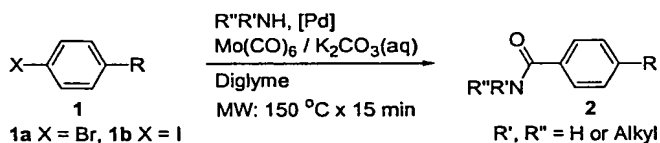
FIG.1



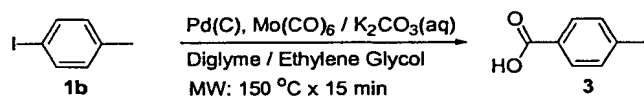


REPLACEMENT SHEET

FIG.2A



Microwave-assisted palladium-catalyzed amidation utilizing in situ generated carbon monoxide from Mo(CO)_6 .



Microwave-assisted palladium-catalyzed generation of *p*-methyl benzoic acid from tolyl iodide utilizing in situ generated carbon monoxide from $[\text{Mo(CO)}_6]$.

^aAverage isolated yields from 2-3 runs (0.23 mmol scale, SmithSynthesizer™, >95% by GC/MS). ^b*p*-Methyl-benzoic acid. Ethylene glycol was added as co-solvent.

entry, aryl-X	R-group	nucleophile	product	yield ^a (%)
1, 1a	MeO-	<i>n</i> -BuNH ₂	2a	70
2, 1a	Me-	<i>n</i> -BuNH ₂	2b	71
3, 1a	F ₃ C-	<i>n</i> -BuNH ₂	2c	75
4, 1a	Ac-	<i>n</i> -BuNH ₂	2d	77
5, 1a	MeO-	Piperidine	2e	65
6, 1a	Me-	Piperidine	2f	66
7, 1a	F ₃ C-	Piperidine	2g	74
8, 1a	Ac-	Piperidine	2h	83
9, 1a	Me-	Benzyl amine	2i	48
10, 1b	MeO-	<i>n</i> -BuNH ₂	2a	69
11, 1b	Me-	<i>n</i> -BuNH ₂	2b	72
12, 1b	F ₃ C-	<i>n</i> -BuNH ₂	2c	78
13, 1b	Ac-	<i>n</i> -BuNH ₂	2d	79
14, 1b	MeO-	Piperidine	2e	66
15, 1b	Me-	Piperidine	2f	69
16, 1b	F ₃ C-	Piperidine	2g	75
17, 1b	Ac-	Piperidine	2h	76
18, 1b	Me-	Water	3	87 ^b



REPLACEMENT SHEET

FIG.2B

4-Acetyl-*N*-*n*-butyl-benzamide (2d). White crystals. ^1H NMR (19 °C, TMS): δ 7.90 (d, 2H; Aryl), 7.77 (d, 2H; Aryl), 6.4 (bs, 1H; CONH), 3.39 (q, 2H; N-CH₂), 2.45 (s, 3H; COCH₃), 1.54 ppm (m, 2H; CH₂), 1.33 (m, 2H; CH₂), 0.89 (t, 3H; CH₃); ^{13}C NMR (CDCl₃, 25 °C, TMS): δ 197 (CO), 166 (CONH), 138,9 (C-ipso), 138,7 (C-ipso), 128 (C-HAryl), 127 (CHAryl), 40 (C-aliphatic), 31 (C-aliphatic), 27 (C-aliphatic), 20 (C-aliphatic), 14 (C-aliphatic). MS (70 eV): m/z (%): 219 (10) [M^+], 177 (25), 147 (100). Elemental Analysis: Calculated for C₁₃H₁₇NO₂: C, 71.2; N, 6.4; H, 7.8; Found: C, 71.6; N, 6.3; H, 7.9 .

4-Trifluoromethylphenyl-piperidin-1-yl-methanone (2g). Yellow oil. ^1H NMR (19 °C, TMS): δ 7.66 (d, 2H; Aryl), 7.48 (d, 2H; Aryl), 3.75 (bs, 2H; CH₂), 3.32 (bs, 2H; CH₂), 1.67 (bs, 4H; CH₂), 1.52 (bs, 2H; CH₂); ^{13}C NMR (25 °C, TMS): δ 168 (CO), 140 (C-ipso), 131 (q; CF₃), 127 (CHAryl), 126 (CHAryl), 122 (C-ipso), 49 (broad, C-aliphatic), 43 (broad, C-aliphatic), 27 (broad, C-aliphatic), 26 (broad, C-aliphatic), 24 (C-aliphatic). MS (70 eV): m/z (%): 256 (80) [M^+-1], 173 (100), 145 (75). Elemental Analysis: Calculated for C₁₃H₁₄F₃NO \times $\frac{1}{2}\text{H}_2\text{O}$: C, 58.6; N, 5.3; H, 5.7; Found: C, 58.8; N, 5.1;



REPLACEMENT SHEET

FIG.3

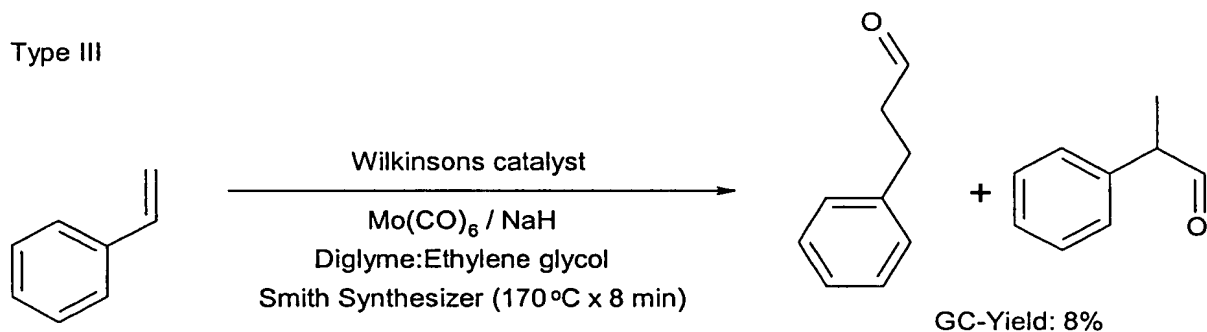
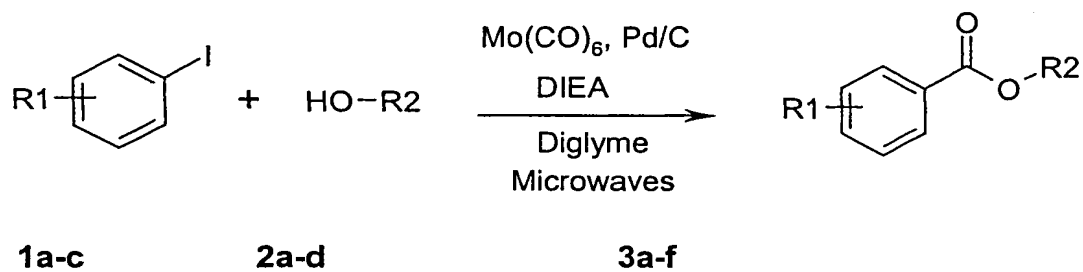


FIG.4



	R1	R2	Time (s)	Conversion of 1a	Isolated Yields (%) of 3
1a	4-OMe	2a -nBu	300	90%	3a 75%
1b	2-Me	2a -nBu	300	Full	3b 46%
1a	4-OMe	2b -tBu	900	Full	3c 38%
1a	4-OMe	2c -CH ₂ Ph	900	b	3d 36%
1a	4-OMe	2d -CH ₂ CH ₂ Si(Me) ₃	900	Full	3e 65%
1c	4-CF ₃	2d -CH ₂ CH ₂ Si(Me) ₃	900	Full	3f 65%

^aMeasured with GC-MS on crude products. ^bnot detected with GC-MS.



REPLACEMENT SHEET

Number	Structure	Name
3a		Butyl 4-methoxybenzoate
3b		Butyl 4-methylbenzoate
3c		t-Butyl 4-methoxybenzoate
3d		Benzyl 4-methoxybenzoate
3e		(2-trimethylsilyl)ethyl 4-methoxybenzoate
3f		(2-trimethylsilyl)ethyl 4-(trifluoromethyl)benzoate

FIG.5



REPLACEMENT SHEET

FIG.6

